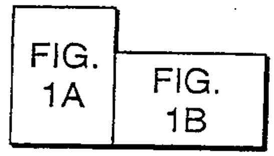


FIG. 1

MORALES, KLR, ETH-SIOH-1 IN CDC13

EXPL S3PUL

SAMPLE	
DATE	FEB 27 98
SOLVENT	CDC 13
FILE	EXP
ACQUISITION	
STFRQ	499.699
TN	111
AT	3.277
NP	39296
SW	5996.1
FB	3400
BS	16
TPWR	63
PW	4.7
DL	0
TOF	0
NL	400
CT	160
ALOCK	N
GAIN	NOT USED
FLAGS	
11	N
LN	N
DP	Y
HS	NN
DISPLAY	
SP	-138.2
WP	5133.1
V$	8848
SC	0
WC	250
NIMM	20.53
LS	33.57
RFL	4131.0
RFP	3627.8
TH	7
INS	1.000
NM	PH

DEC. & VT	
DFRQ	499.699
DN	HL
DPWR	20
DOF	6
DM	NNN
DMM	C
DMF	200
DSEQ	
DRES	1.0
HOMO	N
DEC2	
DFRQ2	0
DN2	
DPWR2	1
DOF2	0
DM2	N
DMM2	C
DMF2	200
DSEQ2	
DRES2	1.0
HOMO2	N
PROCESSING	
16	6.30
WTFILE	
PROC	FT
FN	NOT USED
MATH	R
WERR	
WEXP	
WBS	
WNT	

FIG. 1A

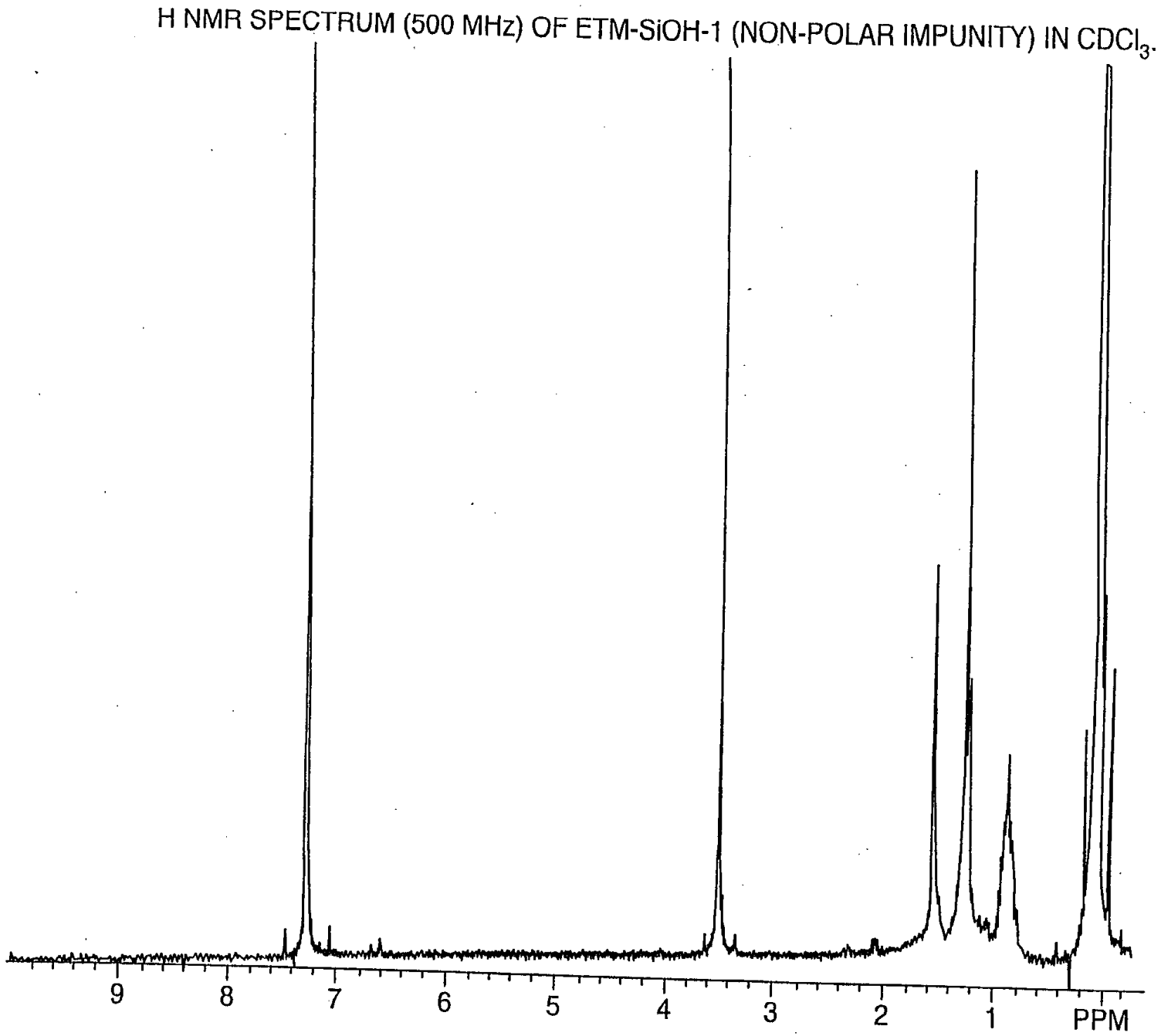
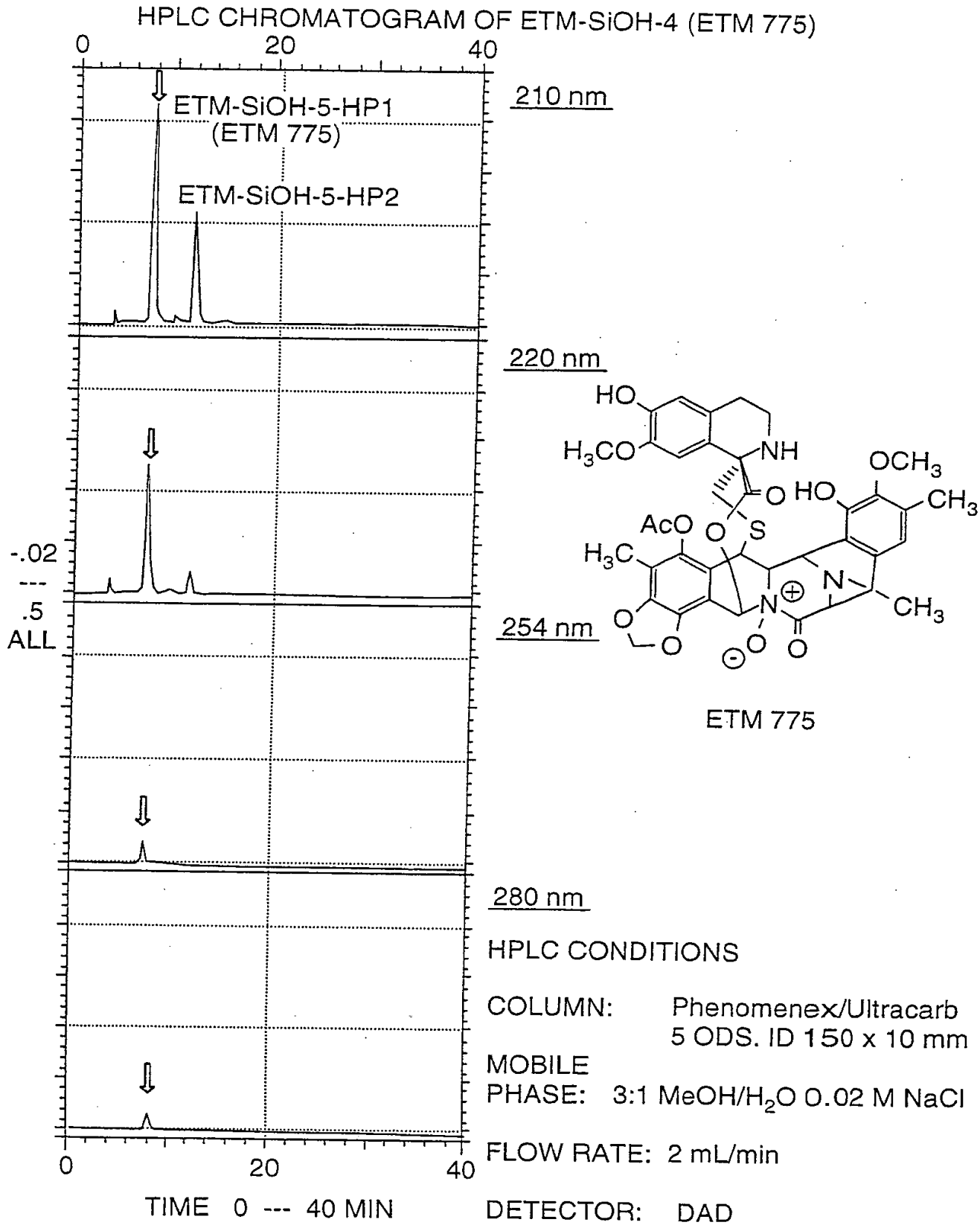

H NMR SPECTRUM (500 MHz) OF ETM-SiOH-1 (NON-POLAR IMPUNITY) IN $CDCl_3$.
9
8
7
6
5
4
3
2
1
PPM

FIG. 1B


HPLC CHROMATOGRAM OF ETM-SiOH-4 (ETM 775)
0
20
40
ETM-SiOH-5-HP1
(ETM 775)
ETM-SiOH-5-HP2
210 nm
220 nm
254 nm
280 nm
-.02

.5
ALL
TIME 0 --- 40 MIN
HPLC CONDITIONS
COLUMN: Phenomenex/Ultracarb
5 ODS. ID 150 x 10 mm
MOBILE
PHASE: 3:1 MeOH/H2O 0.02 M NaCl
FLOW RATE: 2 mL/min
DETECTOR: DAD
ETM 775

FIG. 2

HPLC CHROMATOGRAM OF ETM-SiOH-3 (ETM 305)

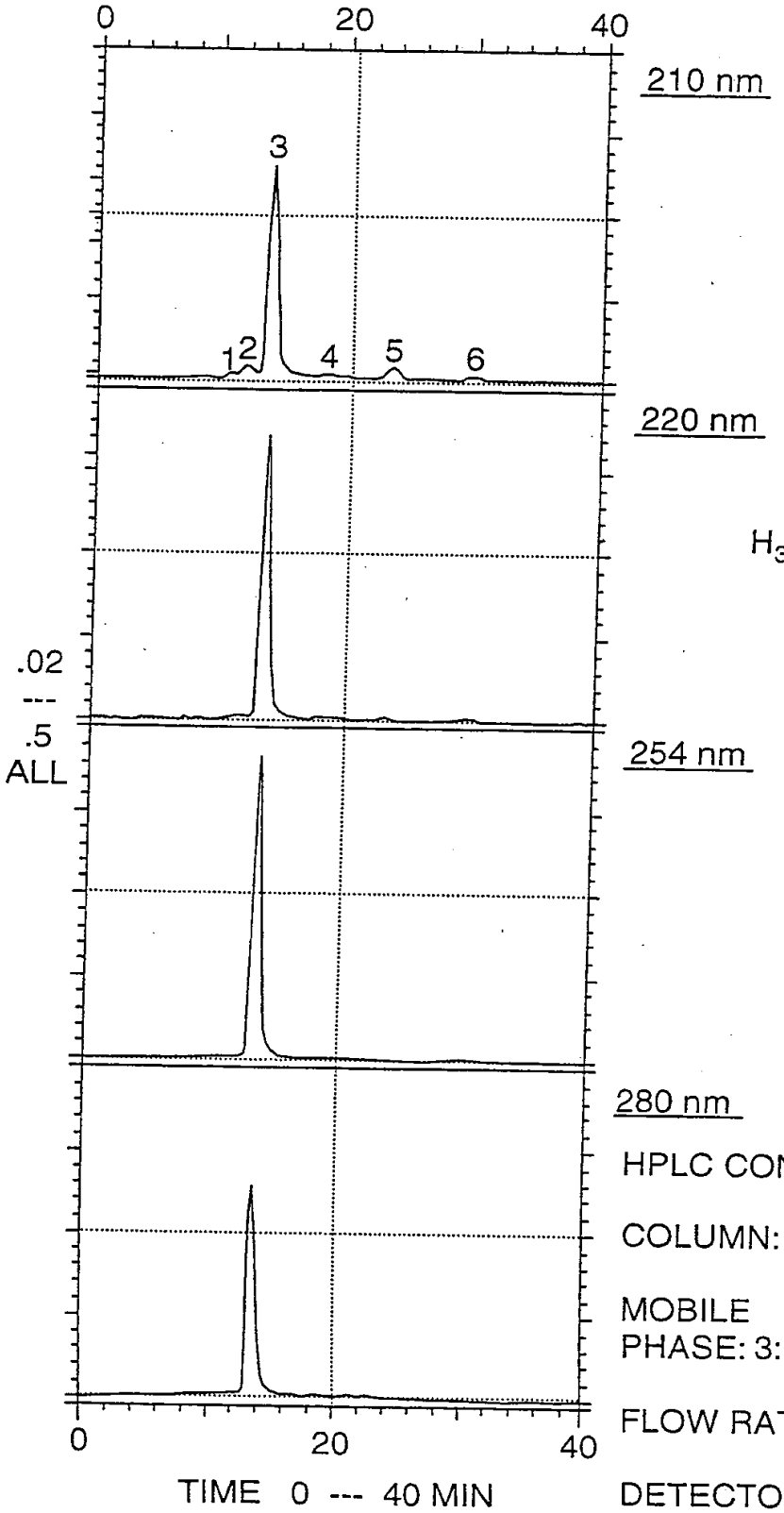
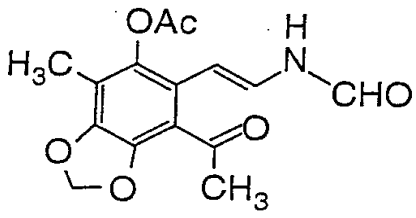


FIG. 3



ETM 305

HPLC CONDITIONS

COLUMN: Phenomenex/Ultracarb 5 ODS. ID 150 x 10 mm

MOBILE PHASE: 3:1 MeOH/H_2O 0.02 M NaCl

FLOW RATE: 1mL/min

DETECTOR: DAD

T0500T 25617660

HPLC CHROMATOGRAM OF ETM-SiOH-2 (TRACE METABOLITES).

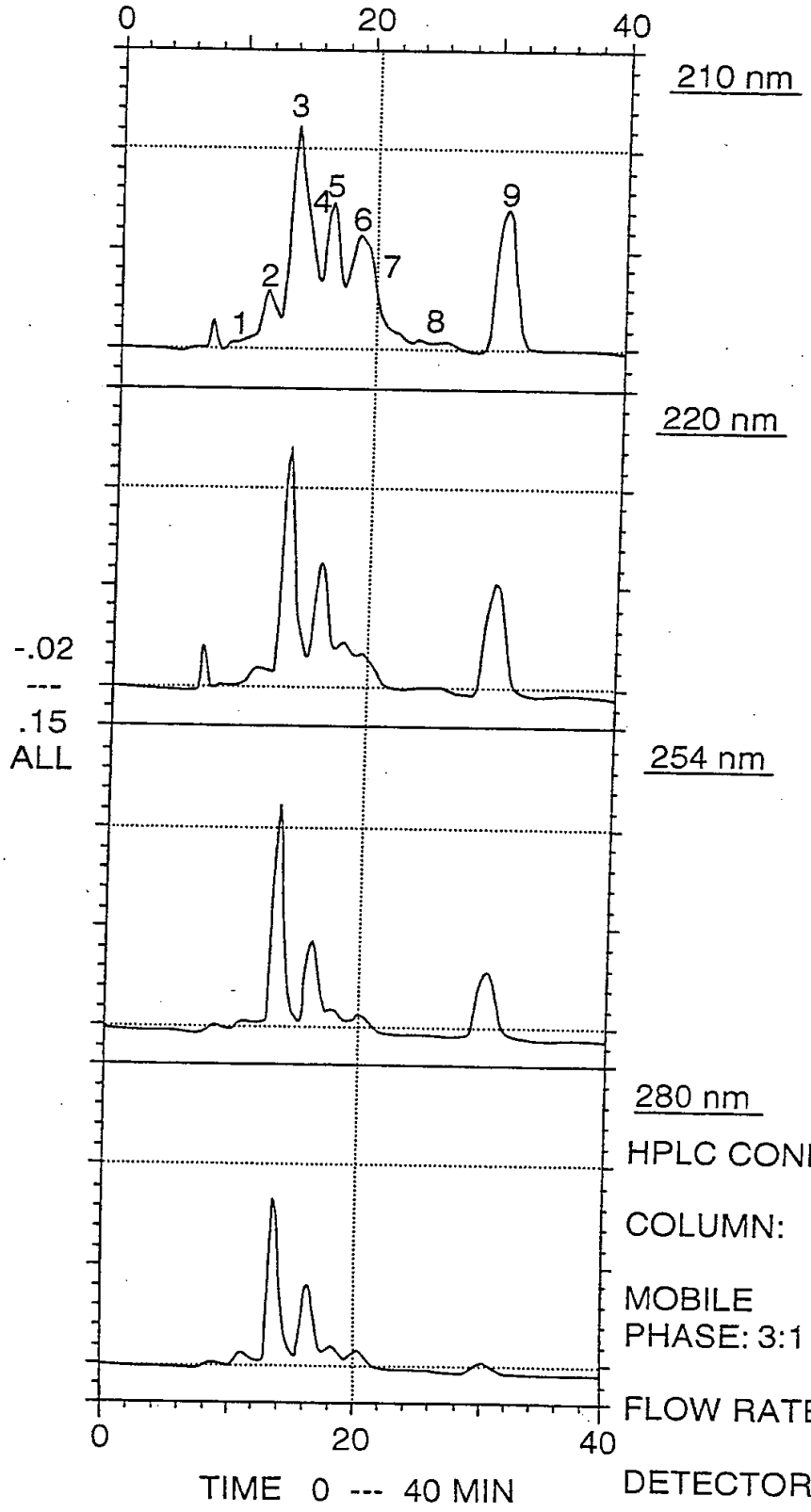


FIG. 4

HPLC CONDITIONS

COLUMN: Phenomenex/Ultracarb 5 ODS. ID 150 x 10 mm

MOBILE PHASE: 3:1 MeOH/H_2O 0.02 M NaCl

FLOW RATE: 1mL/min

DETECTOR: DAD

FOSSIL 25617660

LRFAB MASS SPECTRUM OF ETM 305 IN M.B. (MAGIC BULLET[4])

FILE: ETMSIOH4HP1 IDENT:2 ACQ: 16-DEC-1997 12:43:40 +0:45 CAL:CSI121697
ZAB-SE FAB+ MAGNET BpV: 15.3V TIC:2105487744 FLAGS:HALL
FILE TEXT: MORALES ETM-SIOH-4-HP1 IN M.B.

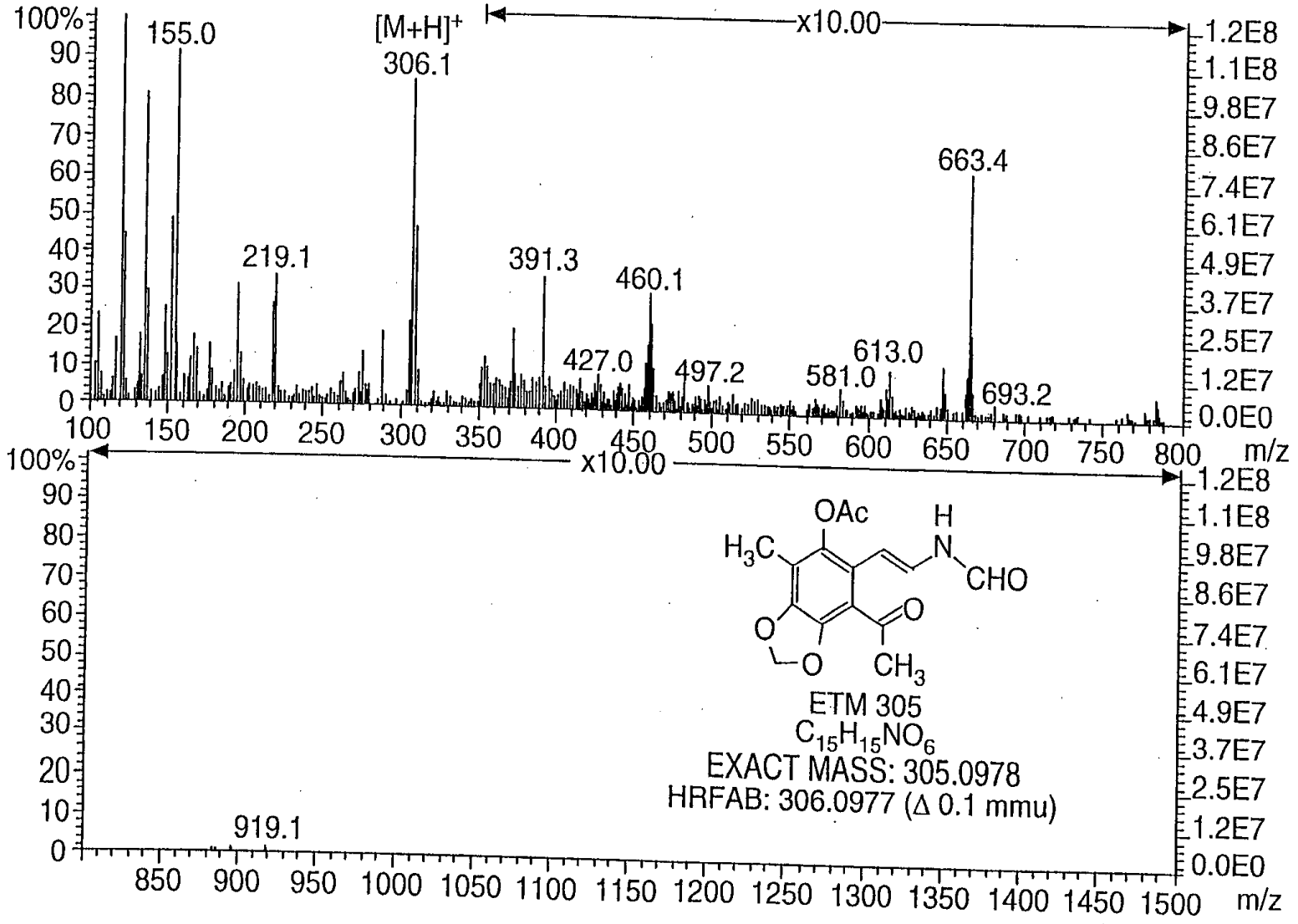


FIG. 5

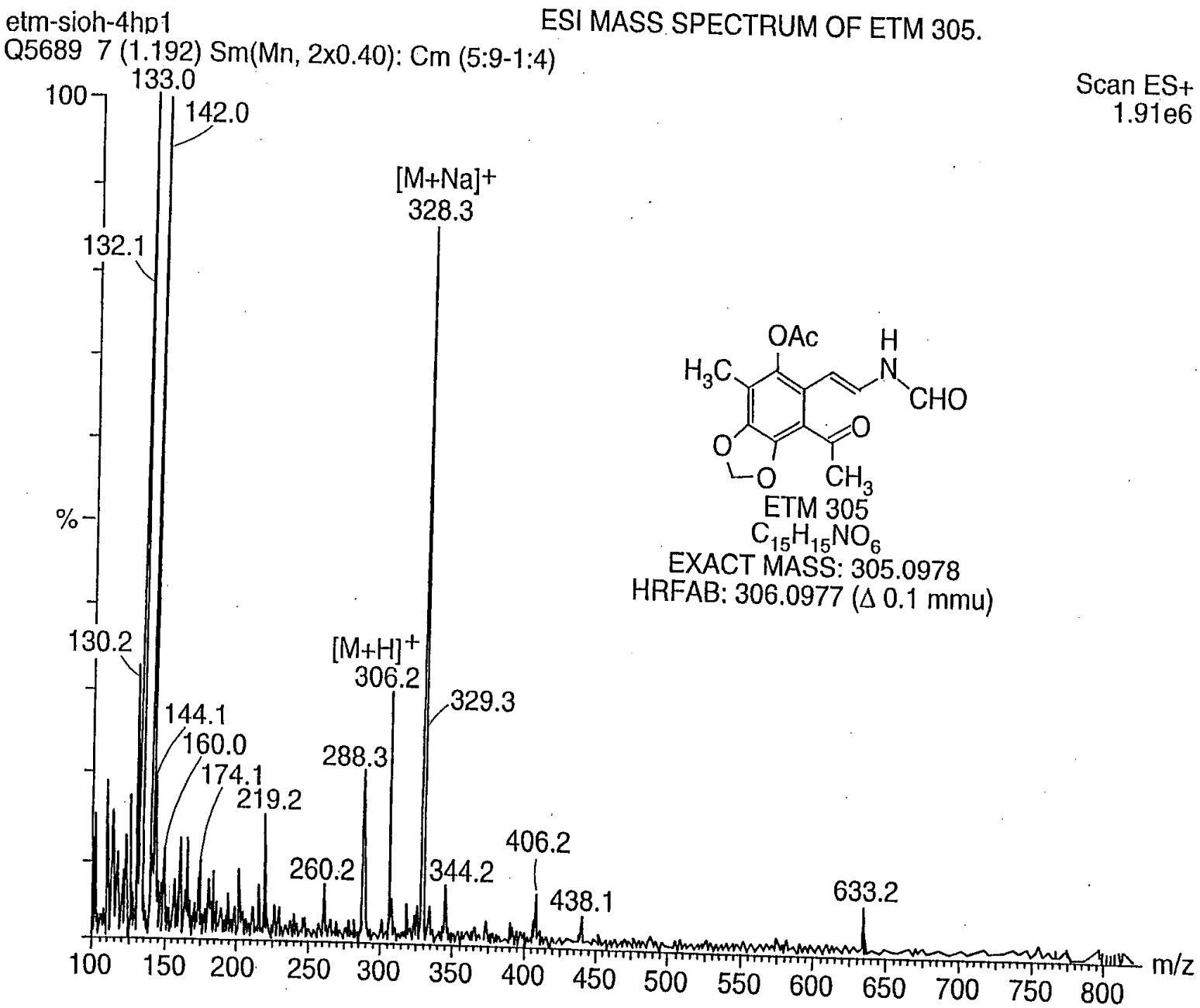
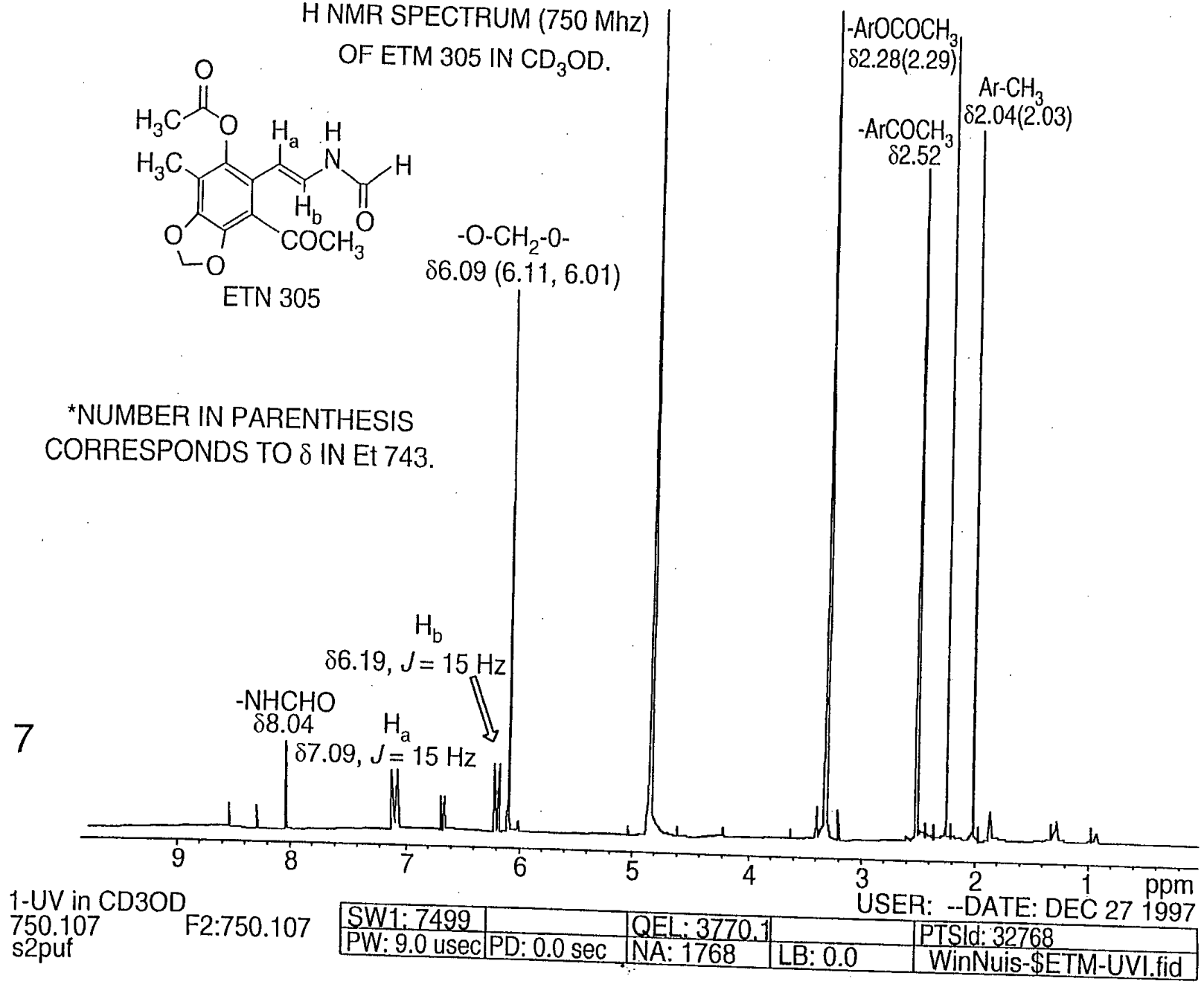

etm-sioh-4hp1
Q5689 7 (1.192) Sm(Mn, 2x0.40): Cm (5:9-1:4)
ESI MASS SPECTRUM OF ETM 305.
Scan ES+
1.91e6
100
%
133.0
142.0
132.1
[M+Na]+
328.3
130.2
[M+H]+
306.2
329.3
144.1
160.0
174.1
219.2
288.3
260.2
344.2
406.2
438.1
633.2
OAc
H
N
CHO
H3C
O
O
O
CH3
ETM 305
C15H15NO6
EXACT MASS: 305.0978
HRFAB: 306.0977 (Δ 0.1 mmu)
100 150 200 250 300 350 400 450 500 550 600 650 700 750 800
m/z

FIG. 6

FIG. 7


H NMR SPECTRUM (750 Mhz)
OF ETM 305 IN CD_3OD.
ETN 305
*NUMBER IN PARENTHESIS
CORRESPONDS TO δ IN Et 743.
-ArOCOCH3
δ2.28(2.29)
Ar-CH3
δ2.04(2.03)
-ArCOCH3
δ2.52
-O-CH2-0-
δ6.09 (6.11, 6.01)
Hb
δ6.19, J = 15 Hz
-NHCHO
δ8.04
Ha
δ7.09, J = 15 Hz
9
8
7
6
5
4
3
2
1
ppm
1-UV in CD3OD
750.107 F2:750.107
s2puf
USER: --DATE: DEC 27 1997
SW1: 7499
QEL: 3770.1
PTSld: 32768
PW: 9.0 usec
PD: 0.0 sec
NA: 1768
LB: 0.0
WinNuis-$ETM-UVI.fid

FAB/MS/MS SPECTRUM OF ETM 305.

FILE:MS9289 IDENT:1 SMO(1,7) PKD(7,3,7,0.00%,0.0,0.00%,F,F) SPEC(HEIGHTS, CENTROID) ACQ:13-FEB-1>
70-4SE FAB+ E2B2 BpM:306 BpV:31.1V TIC:124301384 FM:306.10 FLAGS:NORM
FILE TEXT: MORALES ETM-UV MSMS ON 306.1 CELL 0.5 HE 90% MORALES ETM-UV MSMS ON 306.1 CELL 0.5 HE>

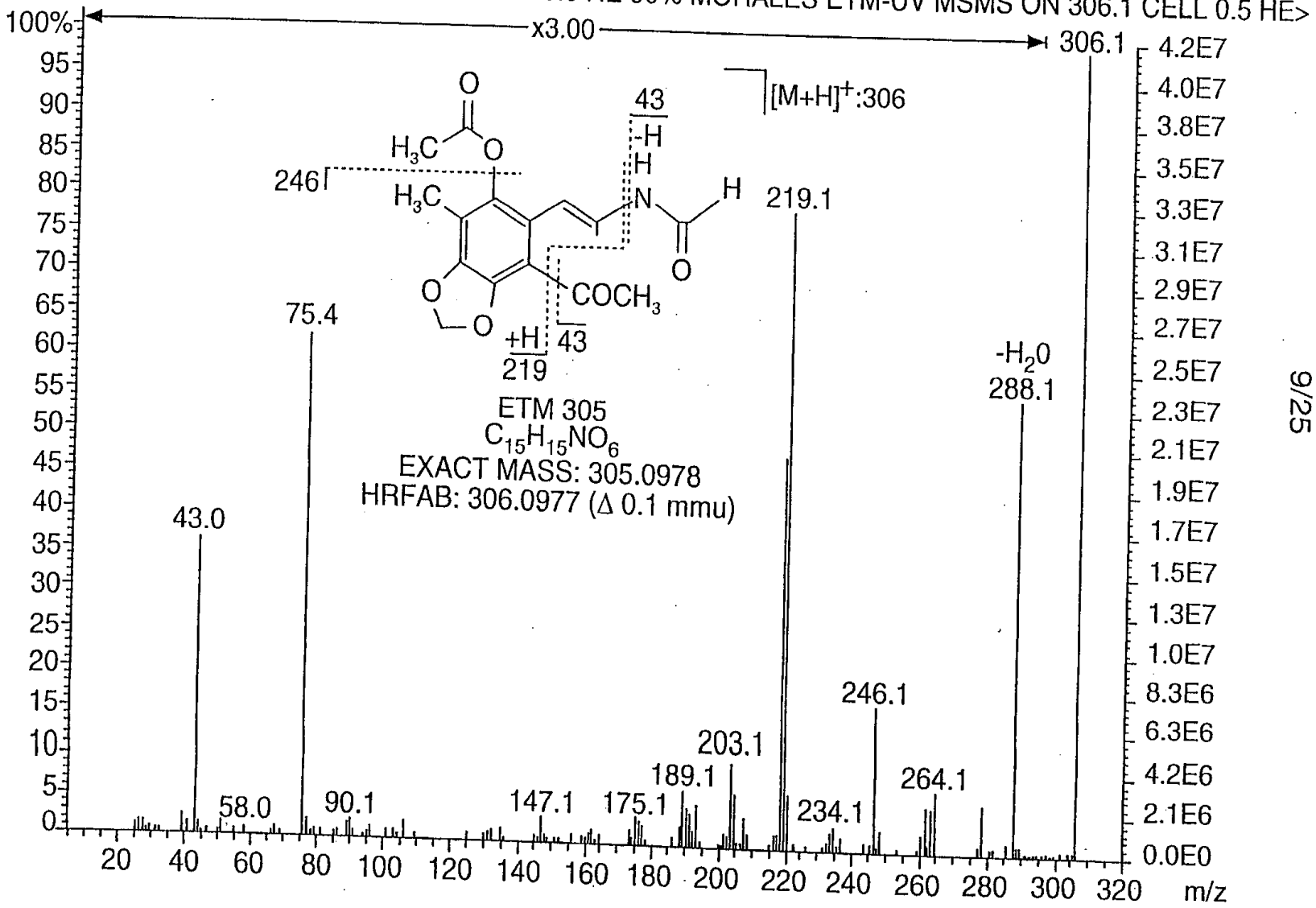


FIG. 8

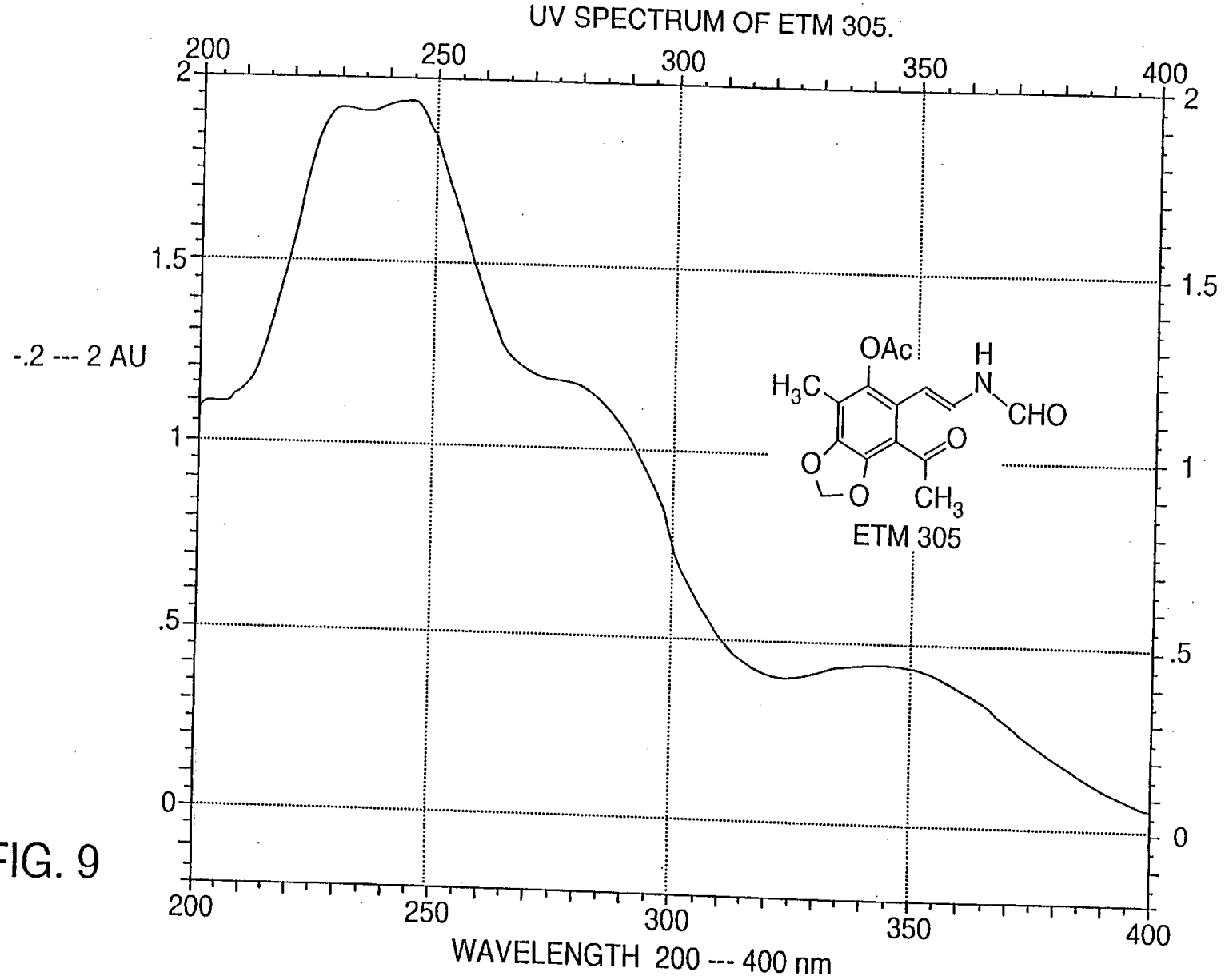

UV SPECTRUM OF ETM 305.
200
250
300
350
400
2
1.5
1
.5
0
-.2 --- 2 AU
OAc
H
N
CHO
H_3C
O
O
O
CH_3
ETM 305
WAVELENGTH 200 --- 400 nm

FIG. 9

UV SPECTRUM OF ETM (PHARMAMAR).

INT OF WINDOW 39: UV APEX SPECTRUM OF PEAK 7.82 OF PICO-M2.D

UV. APEX SPECTRUM OF PEAK 7.82 OF PICO-M2.D

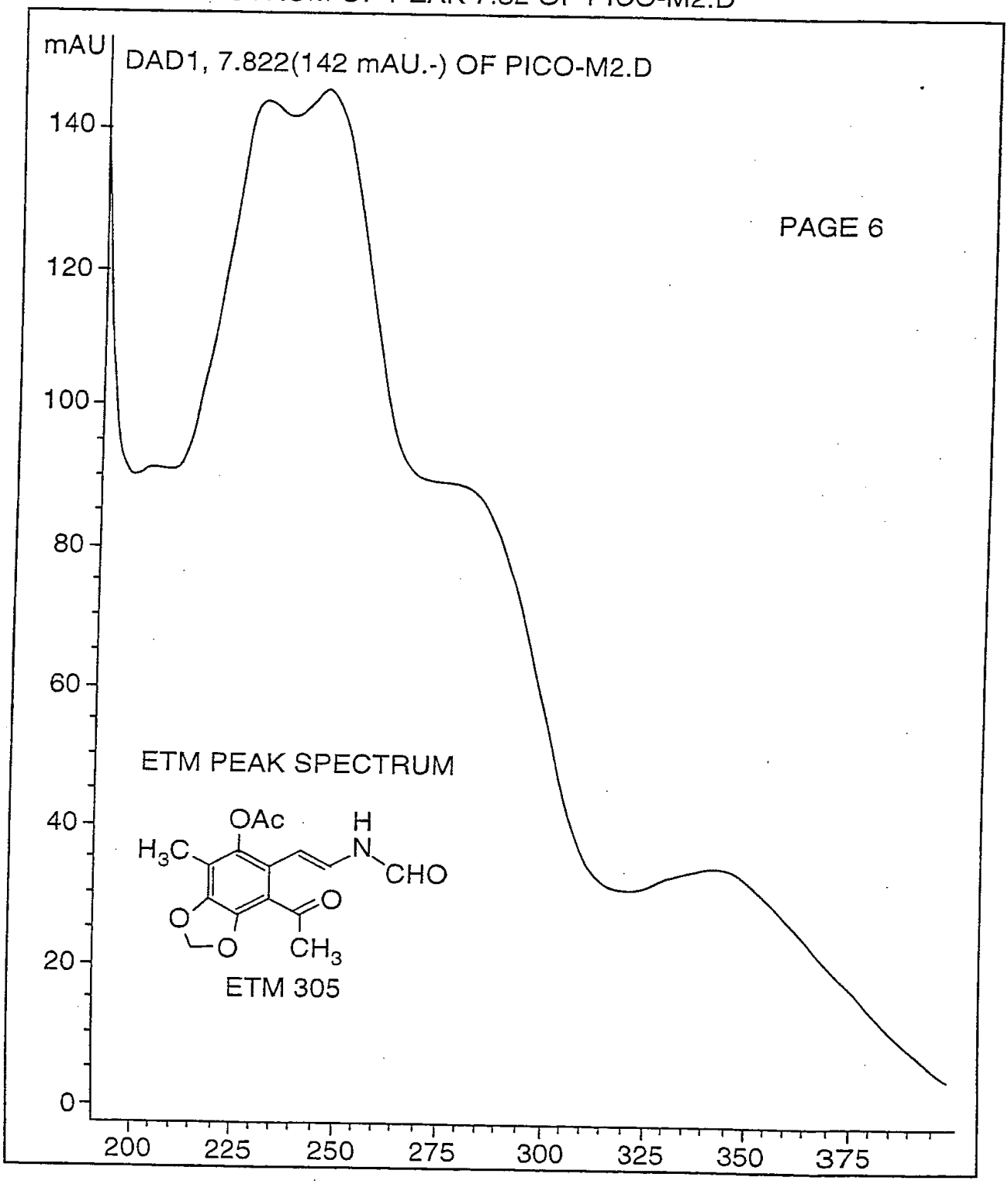


FIG. 10

T0500F-25547650

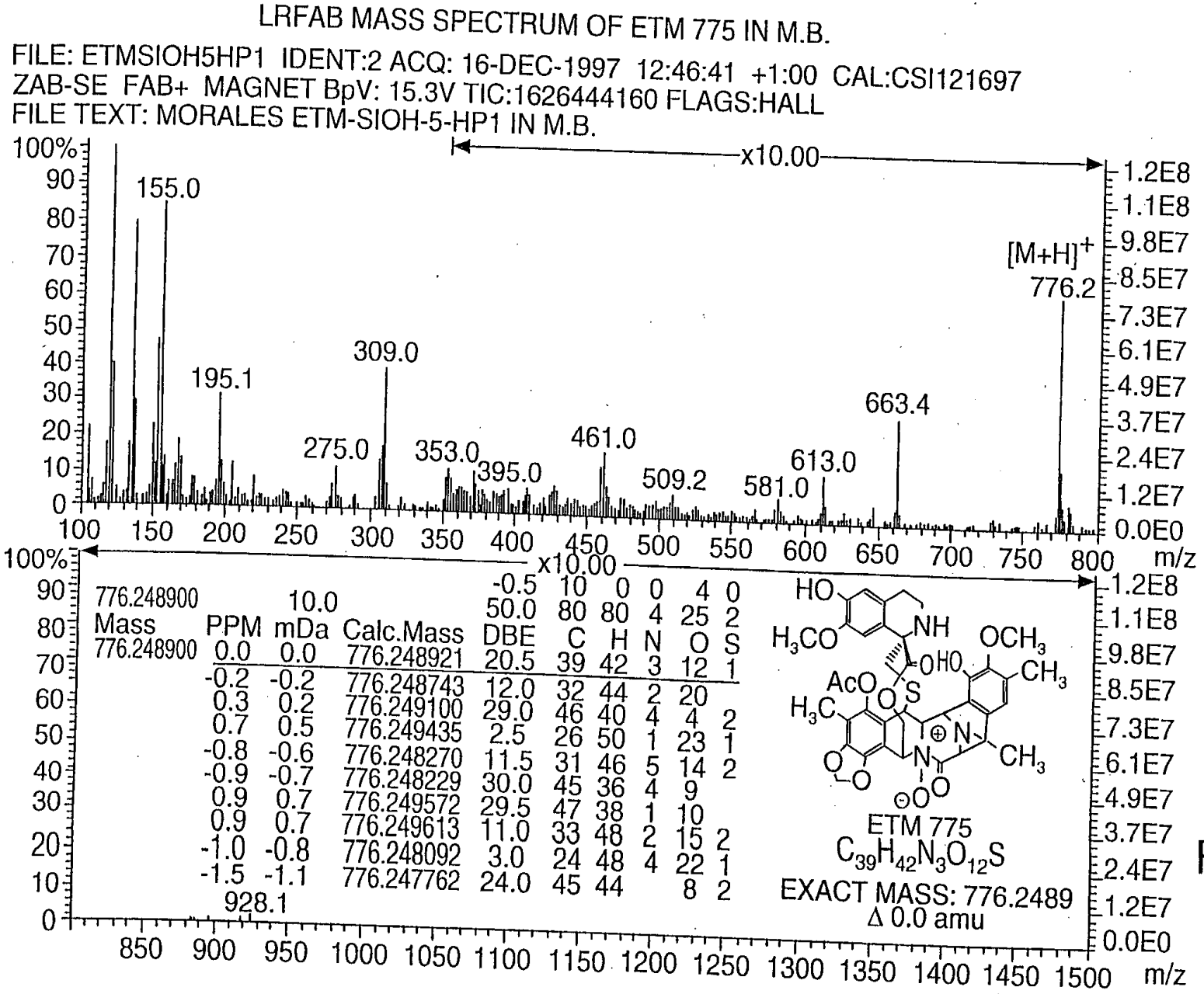

LRFAB MASS SPECTRUM OF ETM 775 IN M.B.
FILE: ETMSIOH5HP1 IDENT:2 ACQ: 16-DEC-1997 12:46:41 +1:00 CAL:CSI121697
ZAB-SE FAB+ MAGNET BpV: 15.3V TIC:1626444160 FLAGS:HALL
FILE TEXT: MORALES ETM-SIOH-5-HP1 IN M.B.
x10.00
[M+H]+
776.2
155.0
309.0
195.1
275.0
353.0
395.0
461.0
509.2
581.0
613.0
663.4
928.1
m/z
776.248900
10.0
Mass PPM mDa Calc.Mass DBE C H N O S
776.248900 0.0 0.0 776.248921 20.5 39 42 3 12 1
-0.2 -0.2 776.248743 12.0 32 44 2 20
0.3 0.2 776.249100 29.0 46 40 4 4 2
0.7 0.5 776.249435 2.5 26 50 1 23 1
-0.8 -0.6 776.248270 11.5 31 46 5 14 2
-0.9 -0.7 776.248229 30.0 45 36 4 9
0.9 0.7 776.249572 29.5 47 38 1 10
0.9 0.7 776.249613 11.0 33 48 2 15 2
-1.0 -0.8 776.248092 3.0 24 48 4 22 1
-1.5 -1.1 776.247762 24.0 45 44 8 2
-0.5 10 0 0 4 0
50.0 80 80 4 25 2
ETM 775
C39H42N3O12S
EXACT MASS: 776.2489
Δ 0.0 amu

FIG. 11

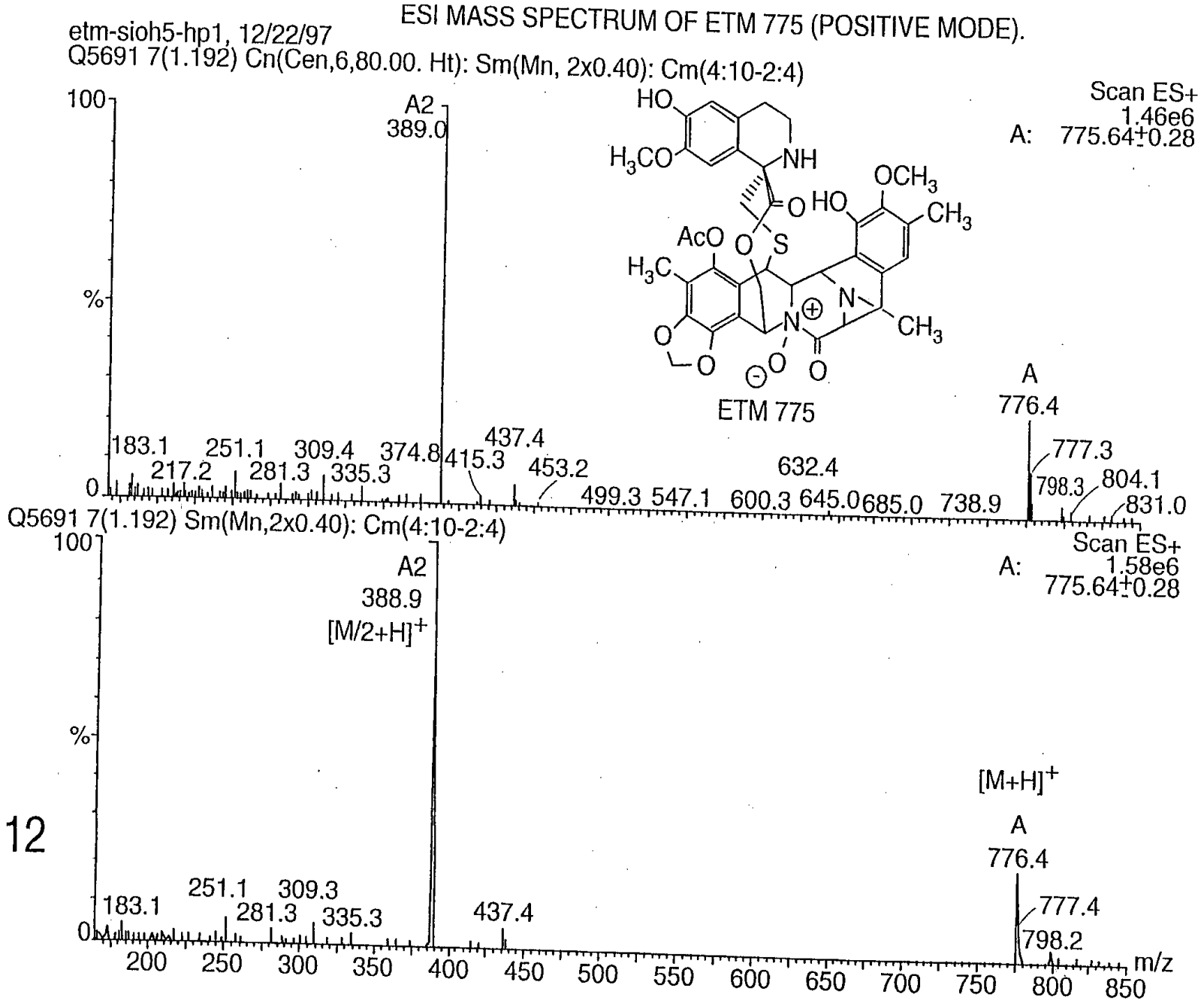

ESI MASS SPECTRUM OF ETM 775 (POSITIVE MODE).
etm-sioh5-hp1, 12/22/97
Q5691 7(1.192) Cn(Cen,6,80.00. Ht): Sm(Mn, 2x0.40): Cm(4:10-2:4)
Scan ES+
1.46e6
A: 775.64±0.28
A2
389.0
ETM 775
A
776.4
777.3
798.3
804.1
831.0
183.1
251.1
309.4
374.8
415.3
437.4
453.2
632.4
499.3
547.1
600.3
645.0
685.0
738.9
217.2
281.3
335.3
Q5691 7(1.192) Sm(Mn,2x0.40): Cm(4:10-2:4)
Scan ES+
1.58e6
A: 775.64±0.28
A2
388.9
[M/2+H]+
[M+H]+
A
776.4
777.4
798.2
183.1
251.1
309.3
281.3
335.3
437.4
100
%
0
200
250
300
350
400
450
500
550
600
650
700
750
800
850
m/z

FIG. 12

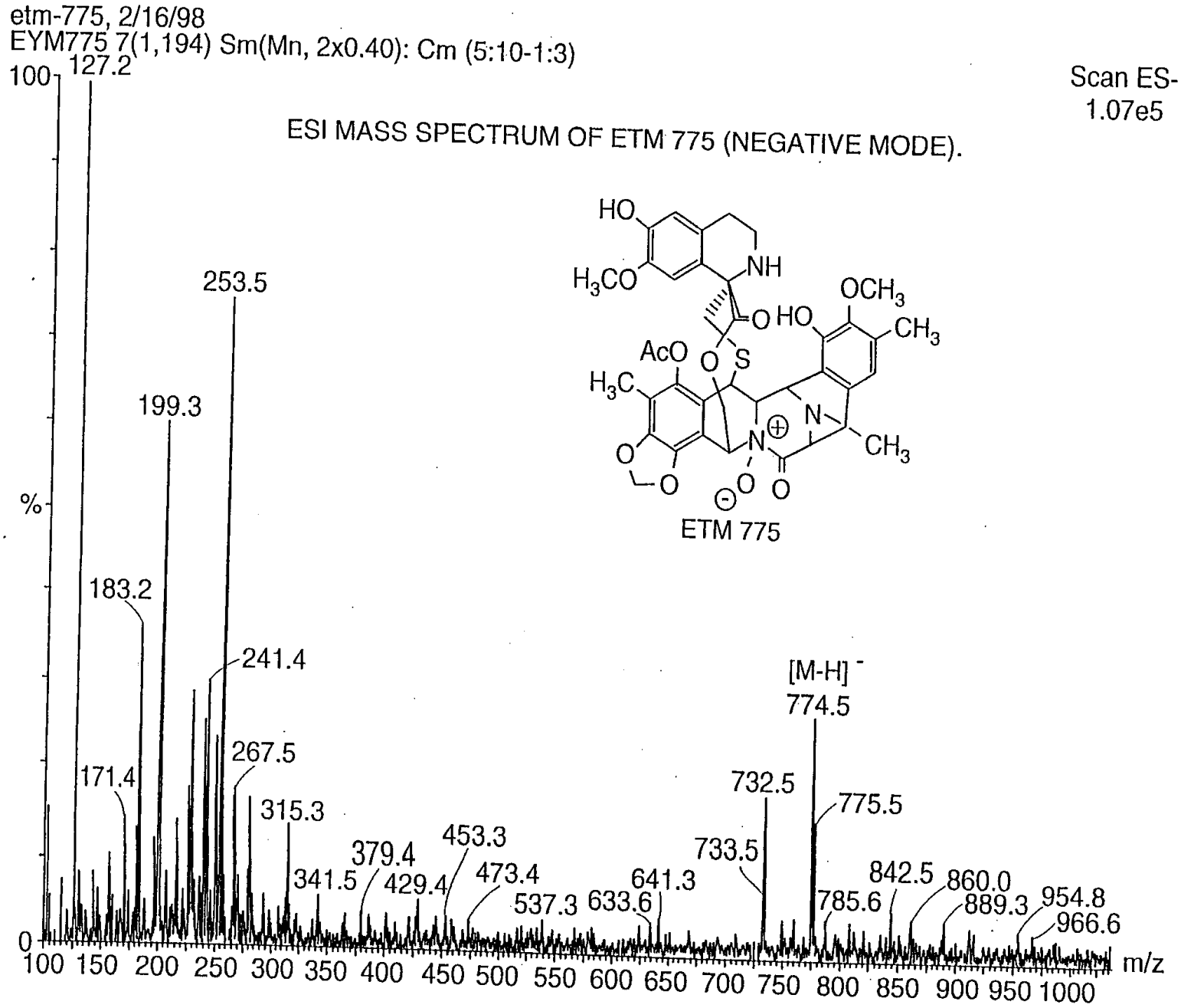

etm-775, 2/16/98
EYM775 7(1,194) Sm(Mn, 2x0.40): Cm (5:10-1:3)
Scan ES-
1.07e5
ESI MASS SPECTRUM OF ETM 775 (NEGATIVE MODE).
ETM 775
100
%
0
127.2
253.5
199.3
183.2
241.4
267.5
171.4
315.3
379.4
341.5
429.4
453.3
473.4
537.3
633.6
641.3
732.5
733.5
774.5
[M-H]⁻
775.5
785.6
842.5
860.0
889.3
954.8
966.6
100 150 200 250 300 350 400 450 500 550 600 650 700 750 800 850 900 950 1000
m/z

FIG. 13

FAB/MS/MS SPECTRUM OF ETM 775 (*m/z* 138-302).

FILE: MS9082 IDENT:1 SMO(1,5) PKD (5,3,5,0.00%,0.0,0.00%,F,F) SPEC (HEIGHTS, CENTROID) ACQ:3-DEC-97>
70-4SE FAB+ E2B2 BpM:777 BpV:146.3V TIC:371164064 FN:776.25 FLAGS:NORM
FILE TEXT: MORALES SFIM2275 MSMS ON 776.25 CELL 0.5 HE 90% MORALES SFIM2275 MSMS ON 776.25 CELL>

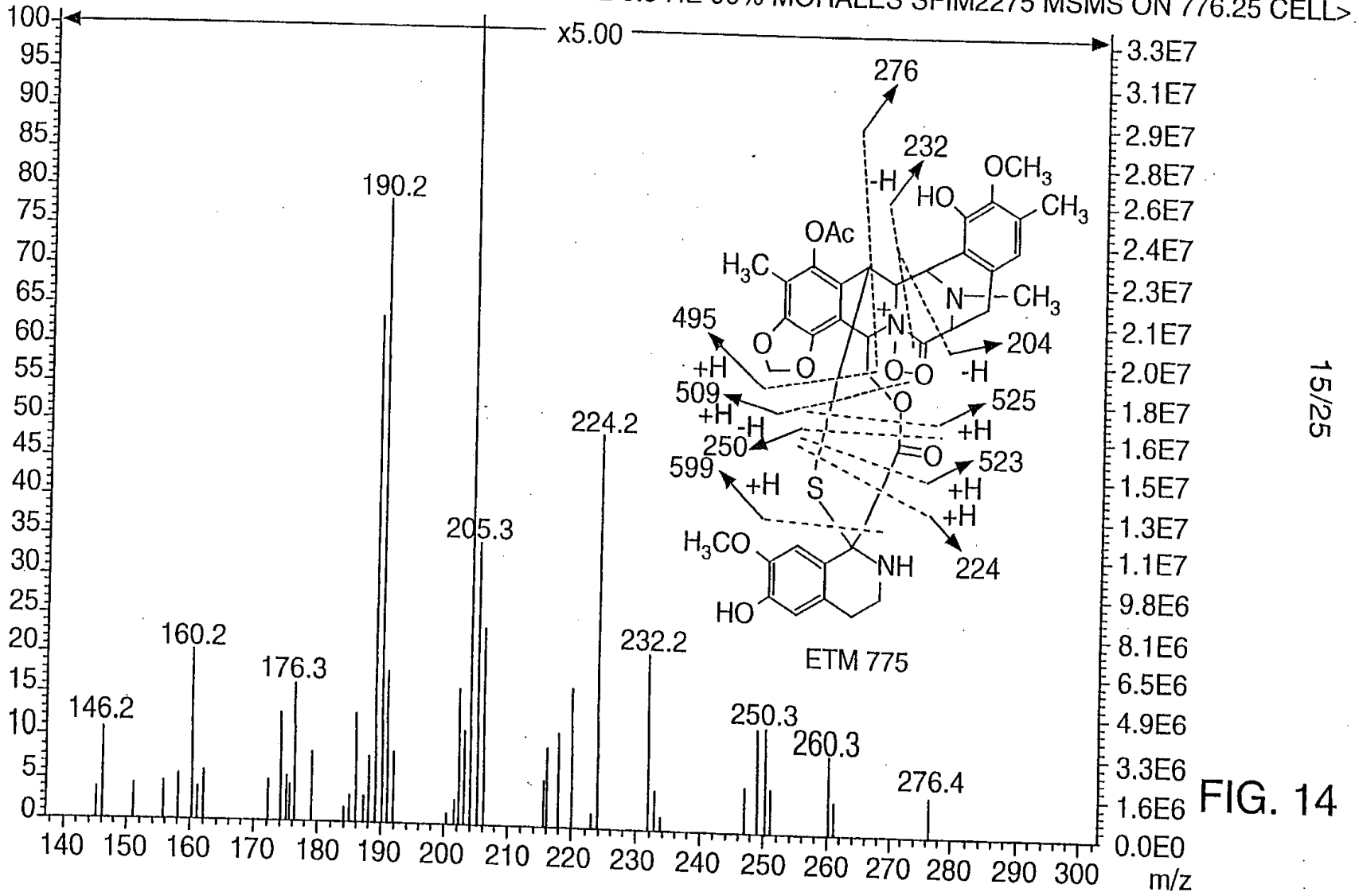


FIG. 14

FAB/MS/MS SPECTRUM OF ETM 775 (*m/z* 440-620).

FILE: MS9082 IDENT:1 SMO(1,5) PKD (5,3,5,0.00%,0.0,0.00%,F,F) SPEC (HEIGHTS, CENTROID) ACQ:3-DEC-97>
70-4SE FAB+ E2B2 BpM:777 BpV:146.3V TIC:371164064 FN:776.25 FLAGS:NORM
FILE TEXT: MORALES SFIM2275 MSMS ON 776.25 CELL 0.5 HE 90% MORALES SFIM2275 MSMS ON 776.25 CELL>

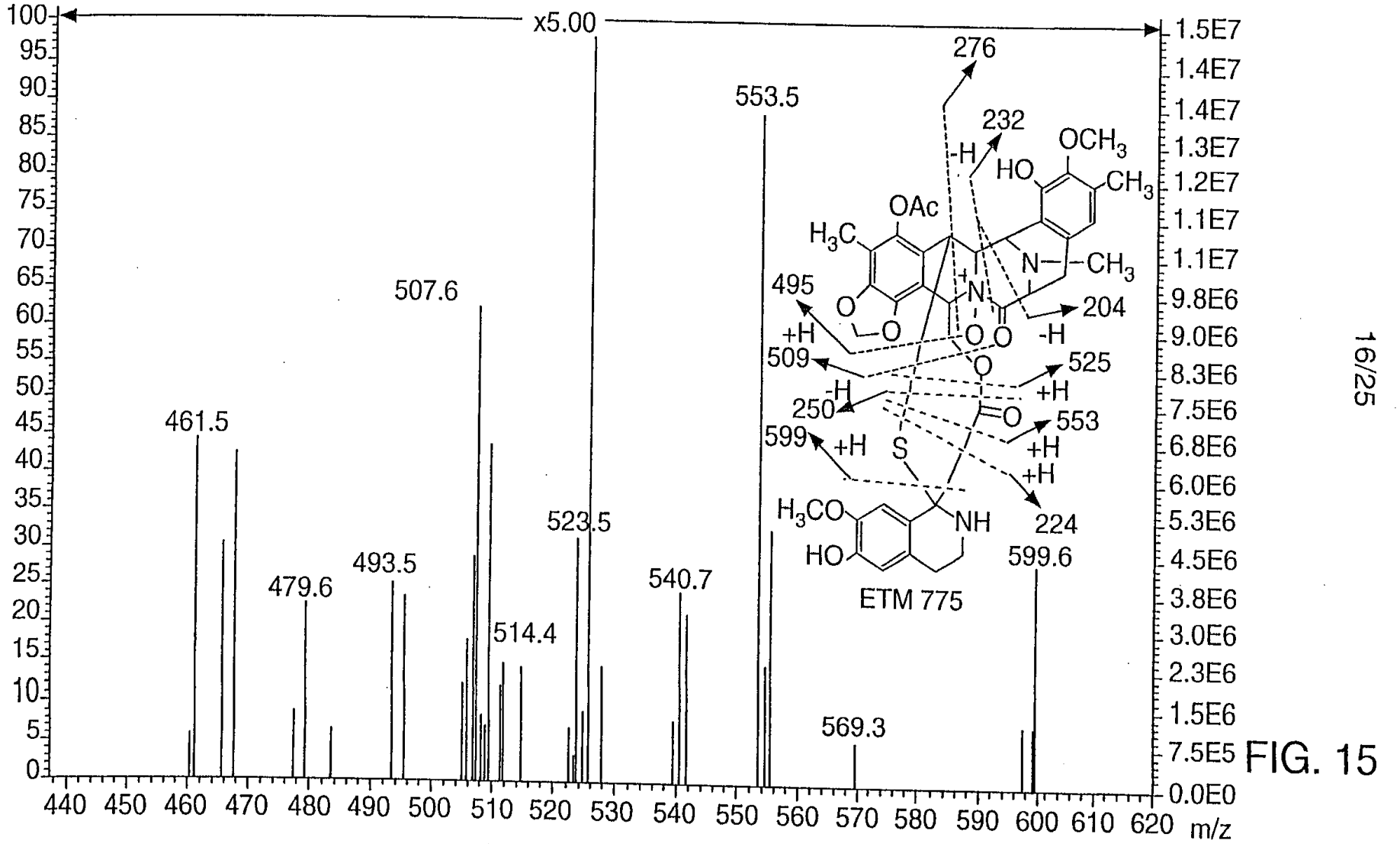


FIG. 15

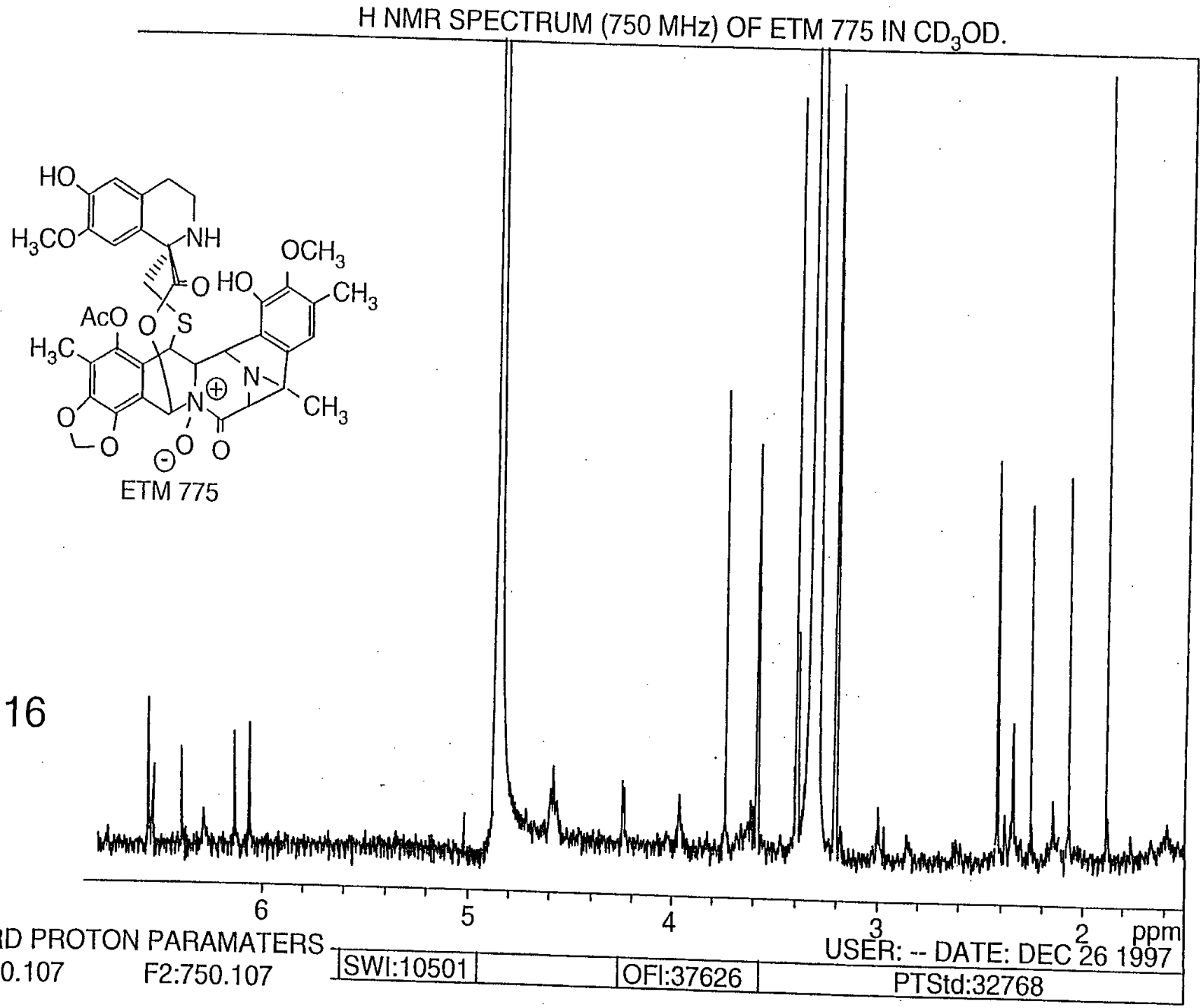

H NMR SPECTRUM (750 MHz) OF ETM 775 IN CD_3OD.
HO
H_3CO
NH
OCH_3
O HO
CH_3
AcO
O
S
H_3C
N
N
CH_3
O
O
O
O
O
ETM 775
6
5
4
3
2
ppm
DARD PROTON PARAMATERS
0.107
F2:750.107
SWI:10501
OFI:37626
USER: -- DATE: DEC 26 1997
PTStd:32768

FIG. 16

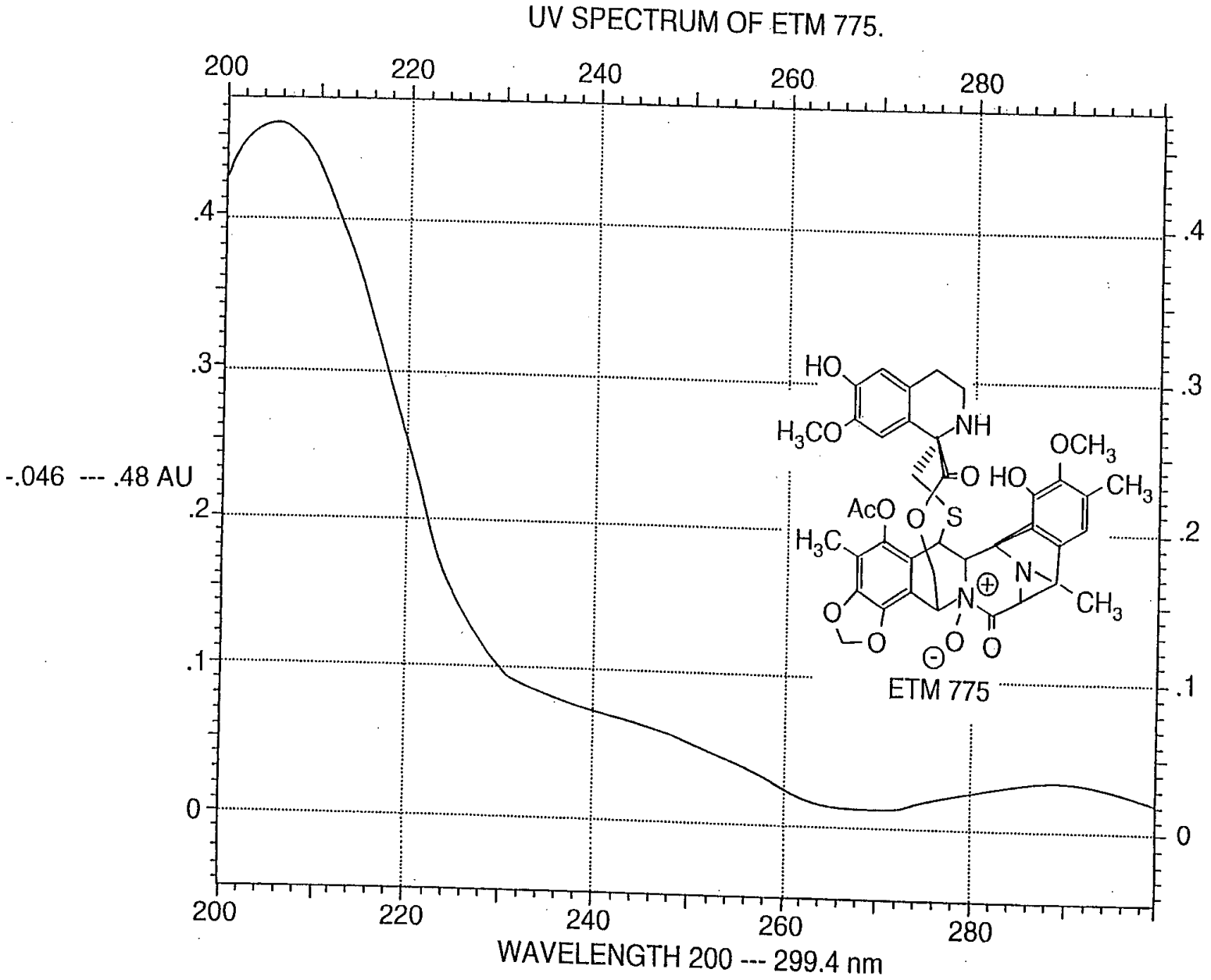
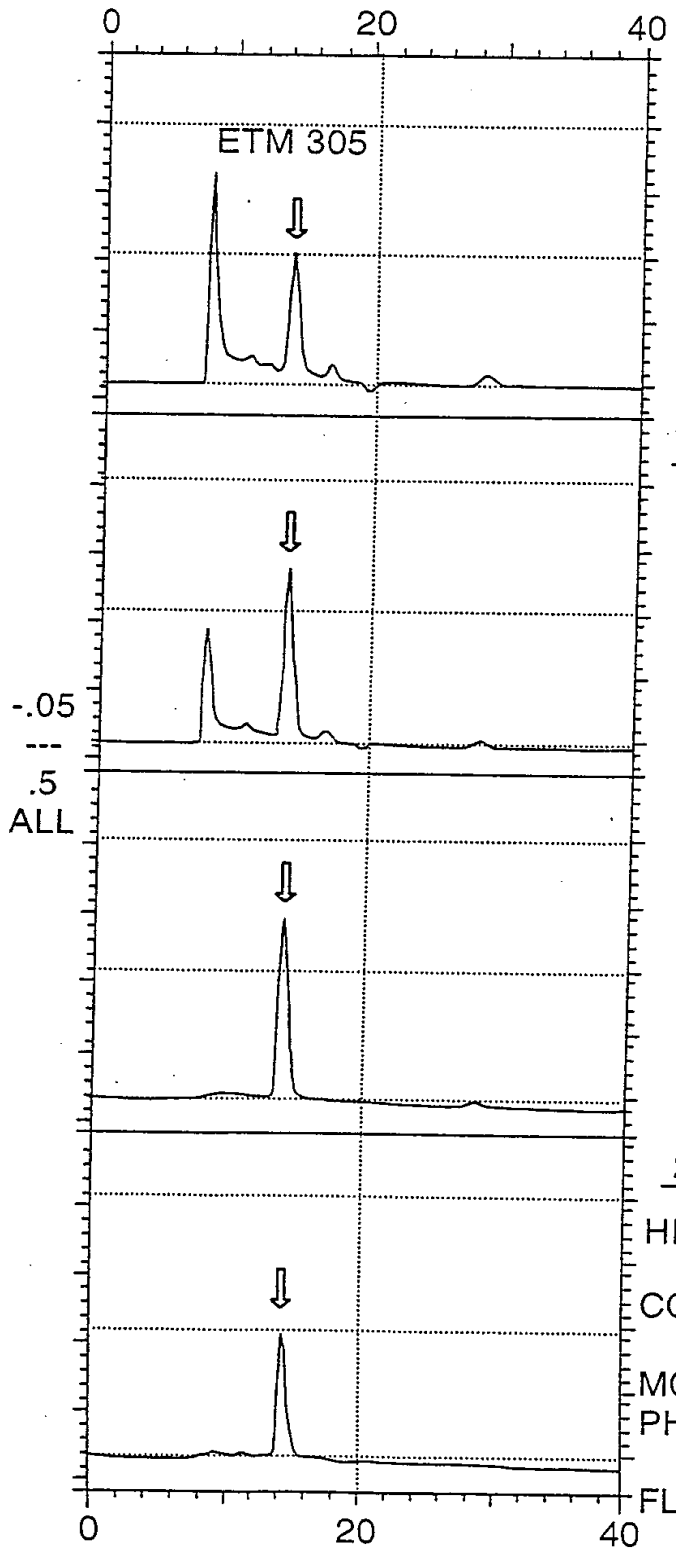
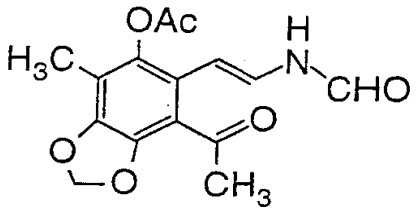

UV SPECTRUM OF ETM 775.
200
220
240
260
280
.4
.3
.2
.1
0
-.046 --- .48 AU
HO
H3CO
NH
OCH3
O HO
CH3
AcO
O
S
H3C
N
N
CH3
O
O
O
O
O
ETM 775
WAVELENGTH 200 --- 299.4 nm

FIG. 17

HPLC CHROMATOGRAM OF M1 METABOLITE (ETM 305).





ETM 305

HPLC CONDITIONS

COLUMN: Phenomenex/Ultracarb 5 ODS. ID 150 x 10 mm

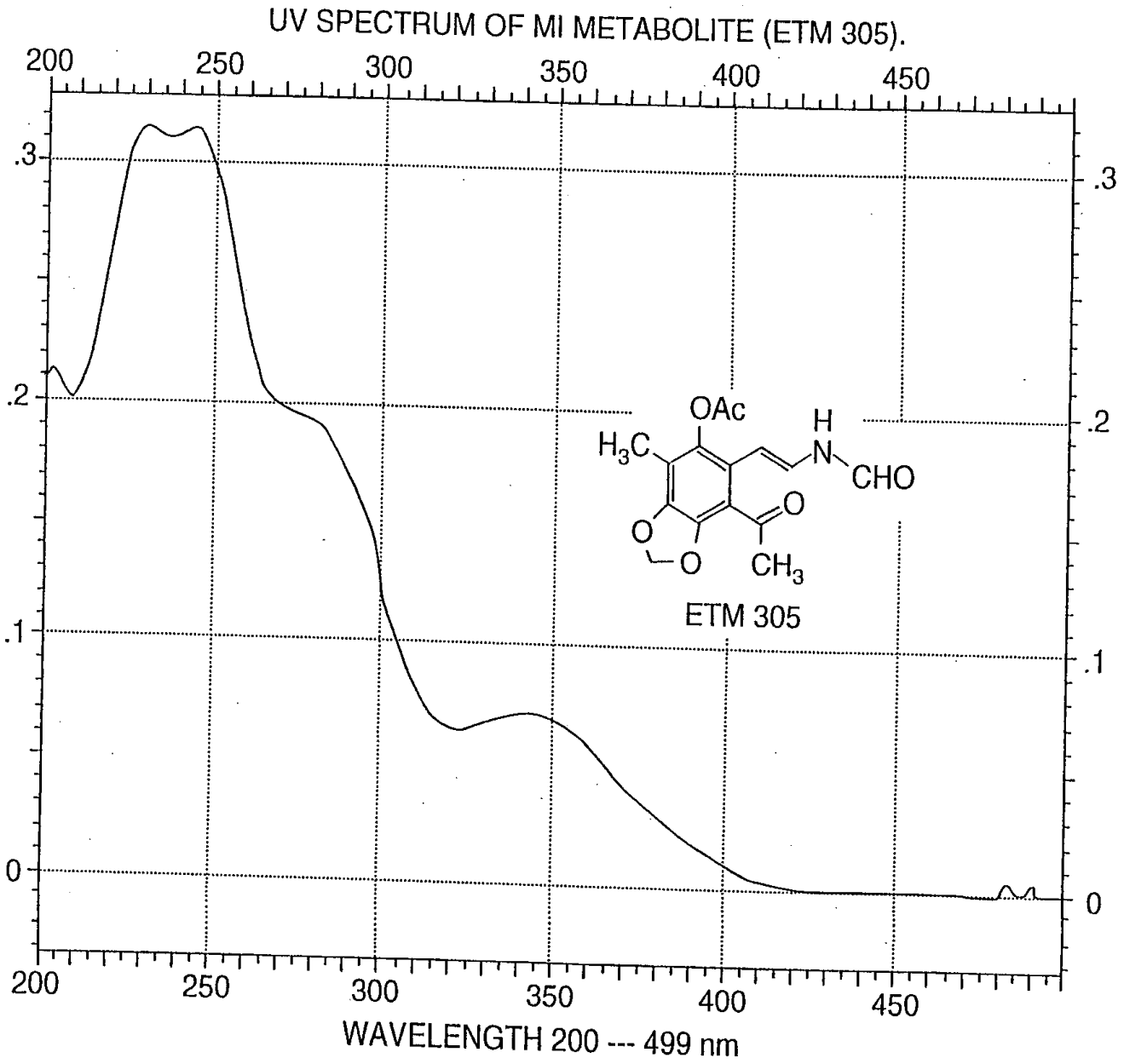
MOBILE PHASE: 3:1 MeOH/H_2O 0.02 M NaCl

FLOW RATE: 1mL/min

DETECTOR: DAD

FIG. 18

T0500F-25F/050



-.033 --- .33 AU

FIG. 19

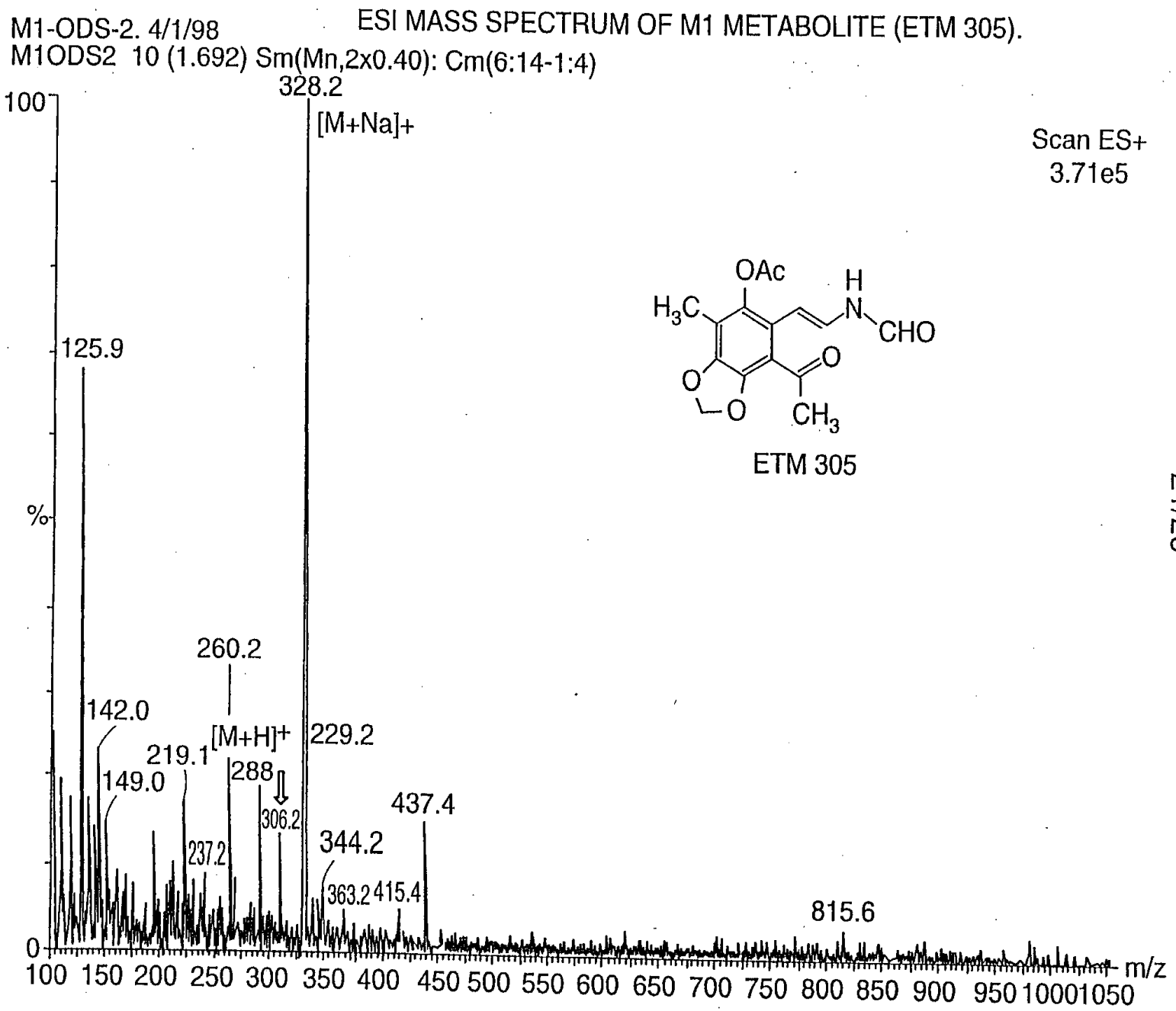

M1-ODS-2. 4/1/98
M1ODS2 10 (1.692) Sm(Mn,2x0.40): Cm(6:14-1:4)
ESI MASS SPECTRUM OF M1 METABOLITE (ETM 305).
Scan ES+
3.71e5
328.2
[M+Na]+
125.9
260.2
142.0
[M+H]+
229.2
219.1
288
149.0
306.2
437.4
237.2
344.2
363.2
415.4
815.6
100
%
0
100 150 200 250 300 350 400 450 500 550 600 650 700 750 800 850 900 950 1000 1050
m/z
OAc
H
N
CHO
H3C
O
O
O
CH3
ETM 305

FIG. 20

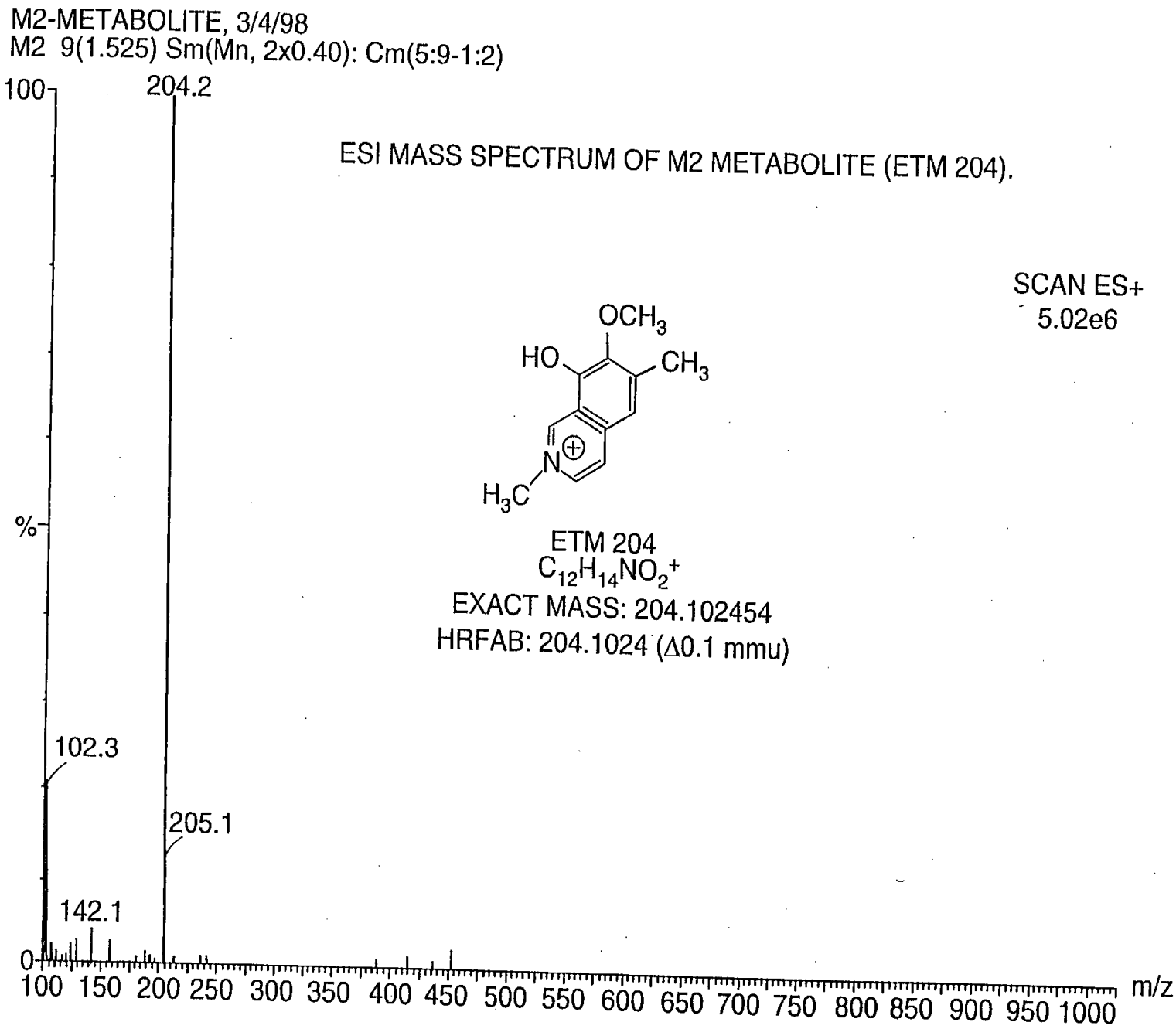

M2-METABOLITE, 3/4/98
M2 9(1.525) Sm(Mn, 2x0.40): Cm(5:9-1:2)
100
204.2
%
102.3
205.1
142.1
0
100 150 200 250 300 350 400 450 500 550 600 650 700 750 800 850 900 950 1000
m/z
ESI MASS SPECTRUM OF M2 METABOLITE (ETM 204).
SCAN ES+
5.02e6
OCH3
HO
CH3
N
H3C
ETM 204
C12H14NO2+
EXACT MASS: 204.102454
HRFAB: 204.1024 (Δ0.1 mmu)

FIG. 21

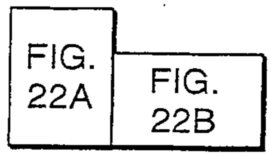


FIG. 22

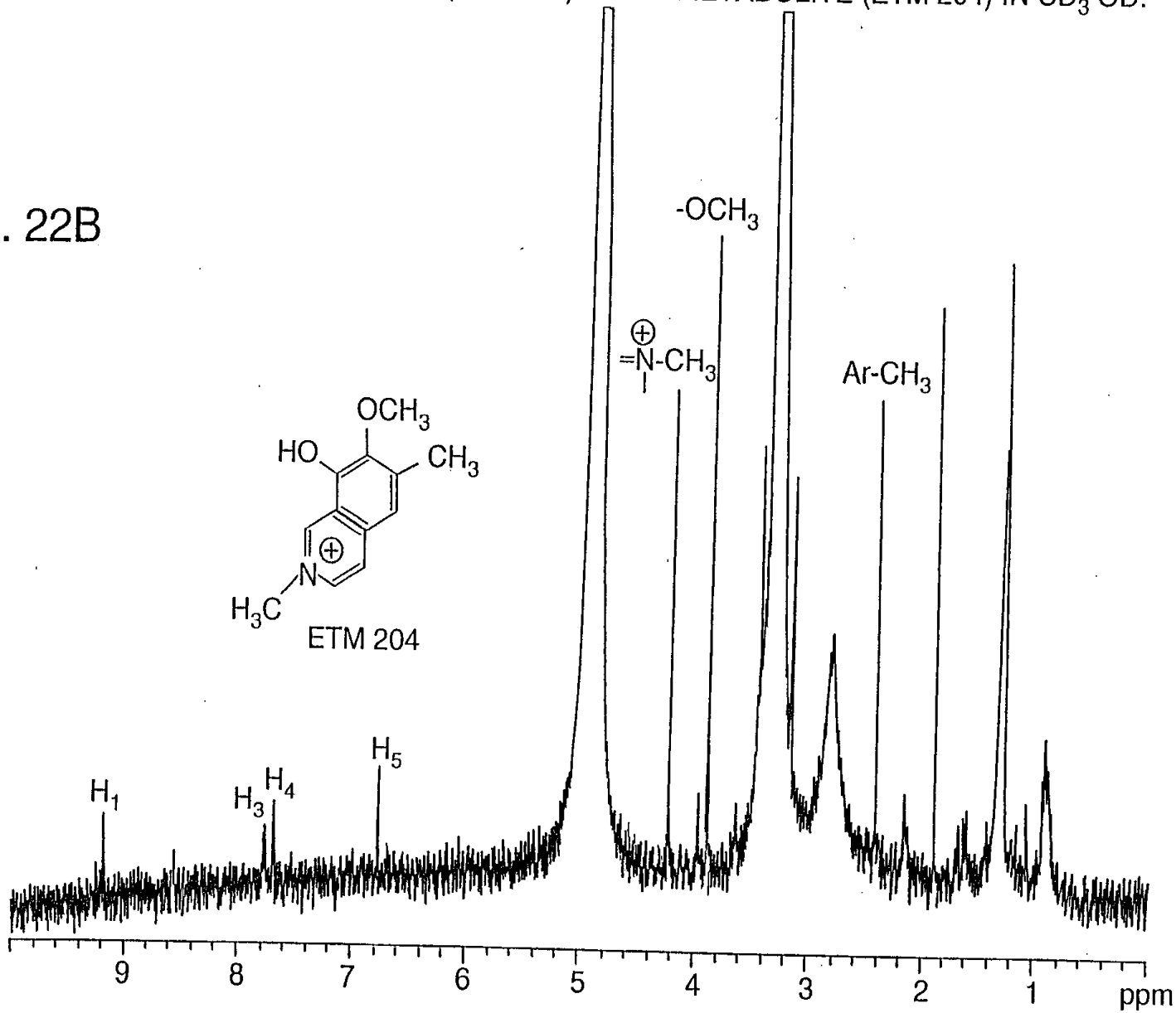
MORALES, KLR, MZ IN CD3OD

EXPL S2PUL

SAMPLE		DEC. & VT	
DATE	MAR 17 98	DFRQ	499.701
SOLVENT	METHANOL	DN	H1
FILE	EXP	DPWR	20
ACQUISITION		DOF	0
STFRQ	499.701	DM	NNN
TN	111	DMM	C
AT	4.003	DMF	200
NP	48000	DSEQ	
SW	5996.1	DRES	1.0
FB	3400	HOMO	N
BS	16	DEC2	
TPWR	63	DFRQ2	0
PW	4.5	DN2	
DL	0	DPWR2	1
TOF	0	DOF2	0
NT	3000	DM2	N
CT	1044	DMM2	C
ALOCK	N	DMF2	200
GAIN	NOT USED	DSEQ2	
FLAGS		DRES2	1.0
11	N	HOMO2	N
LN	N	PROCESSING	
DP	Y	LB	0.30
HS	NN	WTFILE	
DISPLAY		PROC	FT
SP	- 0.1	FN	NOT USED
WP	4997.0	MATH	F
V$	31752		
SC	0	WERR	
WC	250	WEXP	
HZMM	19.99	WBS	
LS	33.57	WNT	
RFL	2154.5		
RFP	1649.0		
TH	7		
INS	1.000		
NM	PH		

FIG. 22A

FIG. 22B


H NMR SPECTRUM (500 MHz) OF M2 METABOLITE (ETM 204) IN CD_3 OD.
-OCH_3
=$\overset{\oplus}{N}$-CH_3
Ar-CH_3
OCH_3
HO
CH_3
H_3C
ETM 204
H_1
H_3
H_4
H_5
9
8
7
6
5
4
3
2
1
ppm

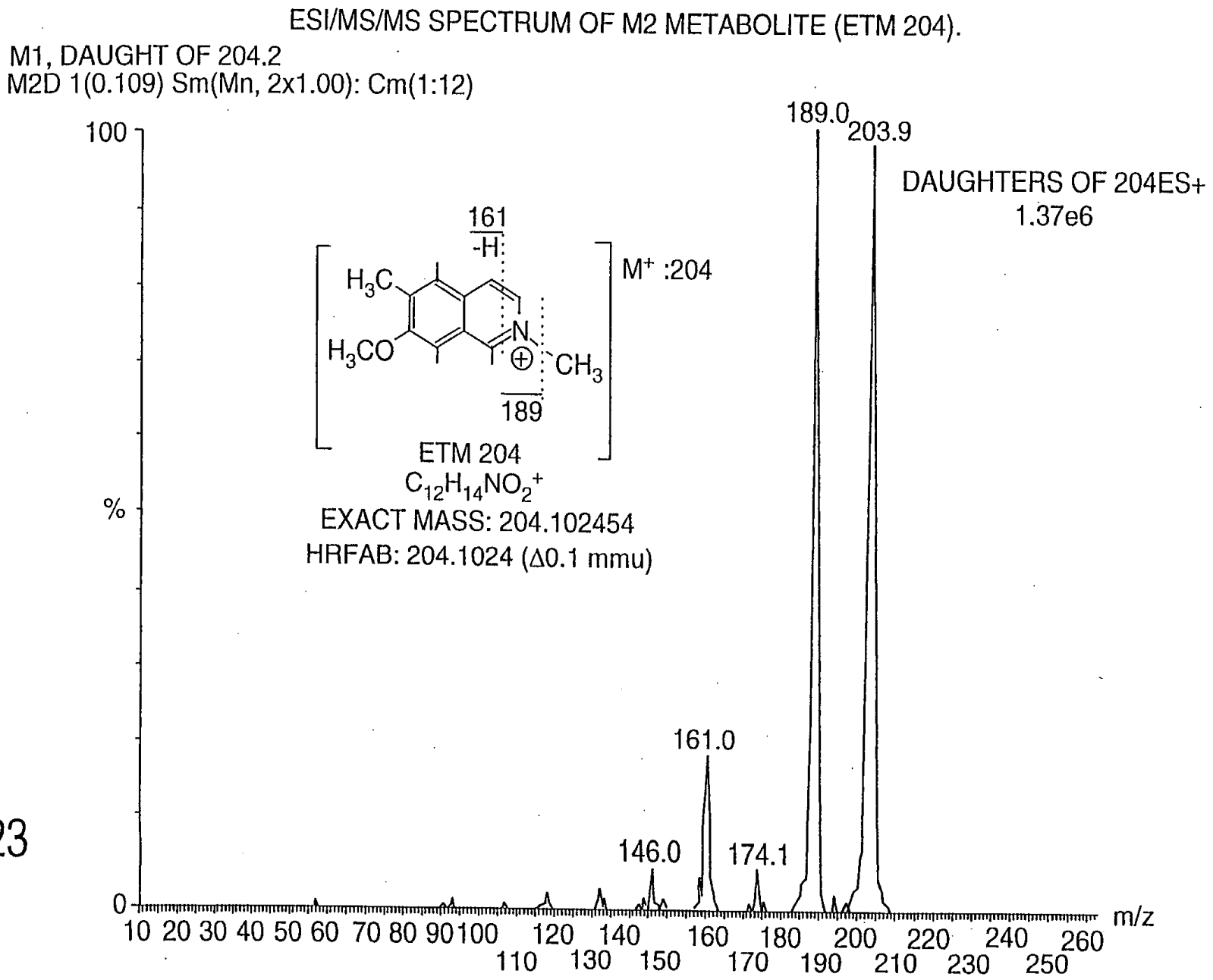

ESI/MS/MS SPECTRUM OF M2 METABOLITE (ETM 204).
M1, DAUGHT OF 204.2
M2D 1(0.109) Sm(Mn, 2x1.00): Cm(1:12)
DAUGHTERS OF 204ES+
1.37e6
189.0
203.9
161.0
146.0
174.1
161
-H
189
M+ :204
ETM 204
C12H14NO2+
EXACT MASS: 204.102454
HRFAB: 204.1024 (Δ0.1 mmu)
100
%
0
m/z
10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190 200 210 220 230 240 250 260

FIG. 23